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POROUS BODIES AND METHOD OF PRODUCTION THEREOF

The present invention relates to porous bodies which are soluble or dispersible in non-aqueous media and to methods of producing such porous bodies.

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Copending international patent application PCT/GB03/03226 (assigned to the present applicants) describes the formation of porous beads comprising a three dimensional open-cell lattice of a water-soluble polymeric material with an average bead diameter in the range 0.2 to 5mm.

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It is an object of the present invention to provide highly porous bodies which dissolve or disperse rapidly when contacted with non aqueous media. It is a further object of the invention to provide a simple and effective method for producing such porous bodies.

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In accordance with a first aspect of the invention, there is provided porous bodies which are soluble or dispersible in non-aqueous media comprising a three dimensional open-cell lattice containing (a) 10 to 95% by weight of a polymeric material which is soluble in water immiscible non-aqueous media and

(b) 5 to 90% by weight of a surfactant,

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said porous bodies having an intrusion volume as measured by mercury porosimetry (as hereinafter described) of at least 3 ml/g

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Preferably the porous bodies of the present invention contain 10 to 80% by weight of the polymeric material and 20 to 90% by weight of the surfactant. More preferably the porous bodies of the present invention contain 20 to 70% by weight of the polymeric material and 30 to 80% by weight of the surfactant.

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It is also important for the operation of the present invention that the porous bodies dissolve or disperse quickly so that the materials contained within the lattice are dispersed quickly when the porous bodies are exposed to a non-aqueous medium. The nature of the lattice should be such that the dispersion of the porous bodies occurs in less than three minutes preferably less than 2 minutes, more preferably less than 30 seconds.

Suitable polymeric materials include homopolymers and copolymers made from one or more of the following (co)monomers:-

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lattice of the porous bodies of the present invention will break down when the porous bodies contact a non-aqueous environment during manufacture releasing the hydrophilic material in a form in which it can be more readily incorporated into the product being manufactured.

- 5 The porous bodies of the present invention may be used to transport materials to sites where they can be incorporated into products. By converting liquid products into porous bodies the need to transport large amounts of liquids can be avoided resulting in significant cost savings and safer transport of materials which are potentially hazardous when transported in a liquid form. Materials which would be potentially unstable if stored or transported in liquid form may be incorporated into
- 10 the porous bodies of the present invention and stored or transported with less risk of degradation.

The incorporation of potentially unstable hydrophilic materials into the porous bodies of the present invention may protect them from degradation during storage prior to use.

- 15 The intrusion volume of the porous bodies as measured by mercury porosimetry (as hereinafter described) is preferably at least about 4 ml/g, even more preferably at least about 5ml/g, and most preferably at least about 6 ml/g. For example, the intrusion volume may be from about 3 ml/g to about 30 ml/g, preferably from about 4 ml/g to about 25ml/g, more preferably from about 7 ml/g to about 20ml/g. Intrusion volume provides a good measure of the pore volume in materials of this
- 20 general type. The polymeric porous bodies may be in the form of powders, beads or moulded bodies. Powders may be prepared by the disintegration of porous bodies in the form of beads or moulded bodies.

- In accordance with another aspect of the present invention, there is provided a method for preparing
- 25 porous bodies which are soluble or dispersible in non-aqueous media comprising a three dimensional open-cell lattice containing
- (a) 10 to 95% by weight of a polymeric material which is soluble in water-immiscible non-aqueous media and
- (b) 5 to 90% by weight of a surfactant,
- 30 said porous bodies having an intrusion volume as measured by mercury porosimetry (as hereinafter described) of at least 3 ml/g
- said process comprising the steps of
- a) providing an intimate mixture of the polymeric material and the surfactant in a liquid medium

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A beaker was placed in a thermostatic vessel and liquid nitrogen was placed in both the beaker and the vessel. The emulsion prepared above was added dropwise from a needle to the liquid nitrogen in the beaker using a A-99 FZ Razel syringe pump. The beaker was placed in a freeze drier overnight to give spherical beads

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Example 3

An experiment was conducted in order to produce highly porous bodies in which the polymeric material is polystyrene (PS). The powder contained about 77% w/w polymer and about 23% w/w surfactant. These bodies were prepared by freezing a water-in-oil emulsion in liquid nitrogen. The emulsion comprised a continuous cyclohexane phase containing PS and a discontinuous aqueous phase. Dioctylsulfosuccinate (AOT) was used as the surfactant.

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A 10% solution of PS was prepared by adding PS (ex Polysciences $M_w = 30000$) to cyclohexane. A sample of the solution (2ml) was stirred with a type RW11 Basic IKA paddle stirrer, and AOT (0.03 g/ml of PS solution) was added followed by water (6ml) to form an emulsion having 75% v/v of discontinuous phase.

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Example 3a

The above emulsion was sprayed into liquid nitrogen from an airbrush. The frozen emulsion was placed in a freeze-drier overnight to give porous bodies in the form of a powder.

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Example 3b

The above emulsion was placed in a beaker which was placed in liquid nitrogen to freeze the emulsion. The frozen emulsion in the beaker was placed in a freeze drier overnight to give a porous moulded body shaped as the inside of the beaker.

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Example 4

An experiment was conducted in order to produce highly porous bodies in which the polymeric material is polystyrene (PS). The powder contained about 77% w/w polymer and about 23% w/w surfactant. These bodies were prepared by freezing a water-in-oil emulsion in liquid nitrogen. The emulsion comprised a continuous cyclohexane phase containing PS and a discontinuous aqueous phase. Dioctylsulfosuccinate (AOT) was used as the surfactant.

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A 10% solution of PS in cyclohexane was prepared by adding PS (ex Polysciences $M_w = 30000$) to cyclohexane. A sample of the solution (2ml) was stirred with a type RW11 Basic IKA paddle stirrer, and AOT (0.06g) was added followed by water (6ml) to form an emulsion having 75% v/v of discontinuous phase. The above emulsion was placed in a beaker which was placed in liquid nitrogen to freeze the emulsion. The frozen emulsion in the beaker was placed in a freeze drier overnight to give a porous moulded body shaped as the inside of the beaker.

The intrusion volume and the bulk density were measured using mercury porosimetry as described above. The dissolution data was determined by taking a sample of the moulded body (0.1g) in cyclohexane (2ml) at 20°C. The results obtained are given in Table 1.

In a similar manner to that described above moulded bodies were prepared. The emulsions from which these bodies were prepared contained PS (2ml - 10 wt% solution in cyclohexane) and AOT (as set out in Table 1 below) and the appropriate volume of water

Table 1

Ex.	Amount of Surfactant (g)	% discontinuous phase	% polymer	% surfactant	Intrusion volume (ml/g)	Dissolution time at 20 °C (min)	Bulk density (g/cm ³)
4	0.06	75	77	23	5.73	0.58	0.14
4a	0.0092	75	96	4		1.42	
4b	0.028	75	88	12		1.63	
4c	0.082	75	71	29		1.67	
4d	0.238	75	46	54		2.58	
4e	0.2	20	50	50		0.42	
4f	0.1	50	67	33		0.75	

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By way of comparison it has been observed that the polystyrene as supplied by the manufacturer had a dissolution time of around 58 minutes.

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and about 3% dye. The bodies were prepared by freezing a water-in-oil emulsion in liquid nitrogen. The emulsion comprised a continuous toluene phase containing polyvinyl acetate and the dye and a discontinuous phase comprising water. Sodium dioctylsulfosuccinate (AOT) was used as the surfactant.

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A 5% solution of polyvinyl acetate in toluene was prepared by adding polyvinyl acetate (PVAc $M_w = 83000$) to toluene. A sample of the solution (2ml) was stirred with a type RW11 Basic IKA paddle stirrer and solvent green 3 dye (0.01g) and AOT (0.25g) were added followed by water (6ml) to form an emulsion having 75% v/v of discontinuous phase.

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Example 8a

The above emulsion was sprayed into liquid nitrogen from an airbrush. The frozen emulsion was placed in a freeze-drier overnight to give porous bodies in the form of a powder.

15 Example 8b

The above emulsion was placed in a beaker which was placed in liquid nitrogen to freeze the emulsion. The frozen emulsion in the beaker was placed in a freeze drier overnight to give a porous moulded body shaped as the inside of the beaker.

20 Example 9

An experiment was conducted in order to produce highly porous bodies containing a hydrophobic dye, which bodies are soluble or dispersible in non-aqueous media in which the polymeric material is polyvinyl acetate. These bodies contained about 33% w/w polymer, about 54% w/w surfactant and 13% dye. The bodies were prepared by freezing a water-in-oil emulsion in liquid nitrogen. The emulsion comprised a continuous toluene phase containing polyvinyl acetate and the dye and a discontinuous phase comprising water. Sodium dioctylsulfosuccinate (AOT) was used as the surfactant.

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30 A 2.5% solution of polyvinyl acetate in toluene was prepared by adding polyvinyl acetate (PVAc $M_w = 83000$) to toluene. A sample of the solution (12ml) was stirred with a type RW11 Basic IKA paddle stirrer and solvent green 3 dye (0.12g) and AOT (0.5g) were added followed by water (12ml) to form an emulsion having 75% v/v of discontinuous phase.

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A beaker was placed in a thermostatic vessel and liquid nitrogen was placed in both the beaker and the vessel. The emulsion prepared above was added dropwise from a needle [Gauge 19] to the liquid nitrogen in the beaker using a A-99 FZ Razel syringe pump. The beaker was placed in a freeze drier overnight to give spherical beads [diameter 2-3mm]

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Example 10

An experiment was conducted in order to produce highly porous bodies containing a hydrophilic dye, which bodies are soluble or dispersible in non-aqueous media in which the polymeric material is polystyrene. These bodies contained about 60% w/w polymer, about 16% w/w surfactant and about 24% w/w dye. The bodies were prepared by freezing a water-in-oil emulsion in liquid nitrogen. The emulsion comprised a continuous cyclohexane phase containing polystyrene and a discontinuous phase comprising water and the dye. Sodium dioctylsulfosuccinate (AOT) was used as the surfactant.

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A 10% solution of polystyrene in cyclohexane was prepared by adding polystyrene (PS ex Polysciences Inc $M_w = 30000$) to cyclohexane. A sample of the solution (2ml) was stirred with a type RW11 Basic IKA paddle stirrer and AOT (0.054g) and an aqueous solution of methyl orange (0.16g) were added to form an emulsion having 50% v/v of discontinuous phase.

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The above emulsion was sprayed into liquid nitrogen from an airbrush. The frozen emulsion was placed in a freeze-drier overnight to give porous bodies in the form of a powder.

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The dissolution time was determined using a sample of the powder (0.1g) in cyclohexane (2ml) at 20°C and was 22 seconds.

Example 11

An experiment was conducted in order to produce highly porous bodies containing a hydrophobic dye, which bodies are soluble or dispersible in non-aqueous media in which the polymeric material is polystyrene. These bodies contained about 60% w/w polymer, about 16% w/w surfactant and about 24% w/w dye. The bodies were prepared by freezing a water-in-oil emulsion in liquid nitrogen. The emulsion comprised a continuous cyclohexane phase containing polystyrene and a discontinuous phase comprising water and the dye. Sodium dioctylsulfosuccinate (AOT) was used as the surfactant.

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